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Single-Lap-Joint Screening of Hysol EA 9309NA Epoxy Adhesive

by Robert E Jensen, David P Flanagan, Daniel C DeSchepper, and Miriam S Silton

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14. ABSTRACT

A commercial 2-part epoxy was evaluated per ARL-ADHES-QA-001.00 rev 1.0, which consists of single-lap-joint testing at room temperature and subsequent hot/wet and elevated temperature conditioning. This testing protocol is intended to provide a minimal recommendation for adhesive selection for Army ground vehicle applications. The epoxy adhesive used for this research is a known aerospace benchmark and was selected to judge the stringency of ARL-ADHES-QA-001.00 rev 1.0, which suggests that the elevated temperature strength retention requirements should be relaxed.

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1. Introduction

The US Army Research Laboratory's (ARL's) screening process for adhesives, per ARL-ADHES-QA-001.00 rev 1.0,[1] is a tiered approach for evaluating bonding performance at a low level of testing effort. The first tier of testing includes collecting relevant materials pedigree information and determining its room temperature (RT) adhesion properties via standardized bonded single lap joints. Materials pedigree information, specifically the reported composition information for hazardous components, is useful for estimating downstream logistical phase-out risk based on current and pending environmental regulations. Second-tier testing consists of further single-lap-joint testing following hot/wet conditioning and at elevated temperature (ET). The adhesive must retain a minimum of 75% of its dry maximum strength (S_{max}) after second-tier testing to be considered for more in-depth testing. Adequate surface pretreatments are required for the adhesive to pass the tiered screening process, particularly for the hot/wet conditioning. For this study a commercial aerospace–grade 2-part epoxy was evaluated in conjunction with traditional silane coupling agent surface pretreatments to judge the stringency of ARL-ADHES-QA-001.00 rev 1.0, particularly with respect to the second tier hot/wet and ET requirements.

2. Experimental

The single lap joints were fabricated and tested using ASTM D1002-10[2] as the basis standard, schematically represented in Fig. 1. Single lap joints are widely studied in the literature and allow for minimal labor-intensive standardized testing.[3]

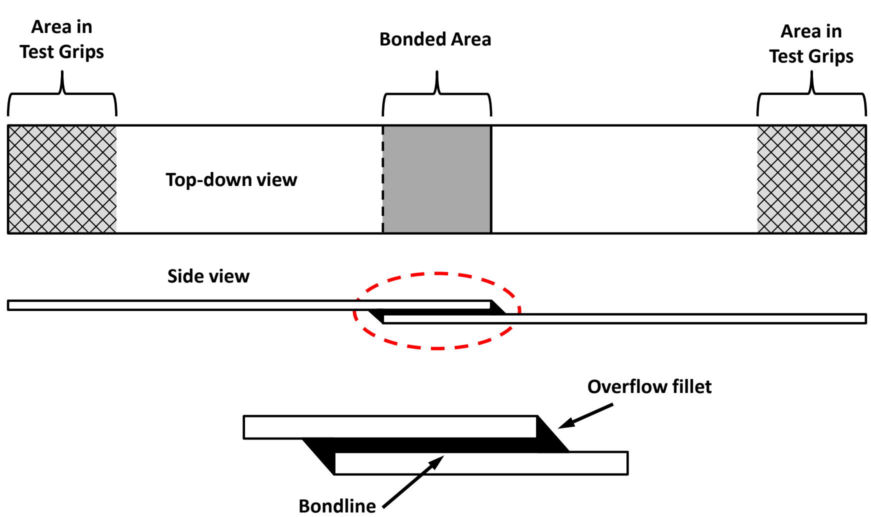

Fig. 1 Adhesively bonded single-lap-joint test specimen configuration (refer to ASTM D1002-10 for dimensions)

Maximum strength (S_{max}) is calculated by dividing the maximum load (P_{max}) by the bonded area (A):

$$S_{max} = \frac{P_{max}}{A} . \tag{1}$$

The maximum strength and mode of failure represent the accepted standard reported outputs of single-lap-joint testing in both industry and academia.[4] The Materials Selection and Analysis Tool (MSAT)[5*] database and the workflow protocols outlined in ARL-TR-7696[6] were used to track the experimental results and analysis.

2.1 Materials and Environmental Assessment

Hysol EA 9309NA epoxy paste adhesive (9309) was used for bonding the single lap joints. Grit blasting and either 3-glycidyloxypropyltrimethoxysilane (GPS) or 3-aminopropyltrimethoxysilane (APS) silane coupling agents were used as the surface pretreatment for the aluminum adherends. Table 1 lists associated environmental restrictions matched against the chemical abstract service (CAS) numbers reported in the manufacturer's safety data sheets of the adhesive and surface pretreatments using the restricted substances database feature of the MSAT database. The highest substance rating returned for current and pending environmental legislation in North America, Europe, and Asia was limited to "caution". None of the constitutive chemical components of the adhesive and surface pretreatments were reported as environmentally "banned".

[*] ARL's MSAT database is housed and supported by NASA Marshall Space Flight Center, Huntsville, AL.

Table 1 Environmental restrictions listed against the adhesive and surface pretreatments

Adhesive	Substance name	CAS no.	Substance rating	Legislation name	Legislation rating
Hysol EA 9309NA	Titanium dioxide	13463-67-7	Caution	ETUC Priority List	Caution
				CoRAP List	Caution
	2,2´-Iminodi(ethylamine)	111-40-0	Caution	ETUC Priority List	Caution
Surface pretreatment					
APS	. . .	. . .	No hazardous substance ratings associated with CAS numbers provided in manufacturer's safety data sheet.	. . .	. . .
GPS	. . .	. . .	No hazardous substance ratings associated with CAS numbers provided in manufacturer's safety data sheet	. . .	. . .
Aluminum oxide grit blast	. . .	1344-24-1	"Caution" for fibrous forms only	. . .	. . .

Notes: APS = 3-aminopropyltrimethoxysilane
CoRAP = European Chemical Agency (ECHA) Community Rolling Action Plan List of Substances
ETUC = European Trade Union Confederation
GPS = 3-glycidyloxypropyltrimethoxysilane

2.2 Sample Preparation

Adhesive bonding of the single lap joints followed procedures outlined in ARL-ADHES-QA-001.01 rev 2.2.[7] 2024 T3 aluminum with an average thickness of approximately 1.62 mm was used for the single-lap-joint substrates. Sandpaper, an acetone wipe-down, and an abrasive pad (3M Scotch-Brite) were used to initially remove oils and large deposits of oxide corrosion from the surface of the aluminum. The coupons were then abrasive media blasted with clean and unused 60-grit aluminum oxide. Residual grit-blasting media was removed by being blown off with a thin stream of nitrogen gas. The APS and GPS silane coupling agent solutions were prepared and applied using a dip-coating technique, as described in ARL-ADHES-QA-001.01 rev 2.2. The pretreated coupons were then heated for 1 h at 100 °C to facilitate condensation and cross-linking of the silane coupling agent to the aluminum oxide surface. Adhesive bonding was completed within 4 h of surface pretreatment.

The 9309 paste adhesive was mixed in a ratio of 100 parts resin to 23 parts curing agent by weight using a FlackTek Inc. (Hauschild Germany) model DAC 400 Speedmixer for 2 min at 2000 revolutions per minute (rpm) followed by 2 min at 2500 rpm. The single lap joints were assembled using the tooling fixture prescribed by ARL-ADHES-QA-001.01 rev 2.2 to provide bondline thickness (0.127 mm shims) and overlap dimension (12.7 × 25.4 mm) control. The adhesive was manually applied to the bonding area using a wooden mixing spatula. Spacer shims were used to set the bondline thickness and contact pressure was maintained by applying weights to the top of the tooling fixture assembly. The adhesive was cured at RT followed by an ET postcure at 66 °C (150 °F) for 1 h. The adhesive was also cast into approximately 3-mm-thick bulk samples using a silicone mold for dynamical mechanical analysis (DMA). Following cure, the single-lap-joint bulk samples were then sanded to remove excess adhesive overflow on the sides to allow for accurate measurement of bondline thickness. Samples requiring RT and ET conditioning were stored under vacuum at RT until testing. Samples requiring hot/wet conditioning were immersed in individual test tubes of deionized water and placed in an oven for 14 days at 63 °C.

2.3 Mechanical Testing

The test procedure for performing the breaking strength of the single lap joint was performed according to paragraphs 9.1 and 9.2 of ASTM D1002-10.[2]

An instrumented mechanical testing frame with a 25-kN load cell was used to ensure that the breaking load of single-lap-joint samples fell between 15% and 85% of the cell's full-scale capacity. A crosshead speed of 1.27 mm/min was used with a pair of self-aligning grips that held the outer 25.4 mm of each end of the single lap joint.

The hot/wet and ET conditions recommended per ARL-ADHES-QA-001.00 rev 1.0 are based on test method standards provided by MIL-STD-810G "Environmental Engineering Considerations and Laboratory Tests".[8,9] Testing of samples with hot/wet conditioning occurred on the same day as their removal from the oven, after allowing them to cool to a reasonable temperature within their respective test tubes. Each sample was patted dry with a paper towel prior to mechanical testing. Samples undergoing ET conditioning were stabilized at 71 °C ± 3 °C for 10 min as measured by an adjacent thermocouple. The heated test chamber used in the test frame was stabilized at 71 °C ± 3 °C for 45 min prior to the start of testing.

The failure surfaces of the broken single-lap-joint coupons were digitally imaged using a flatbed scanner (HP OfficeJet D145). The failed test coupons were manually labeled with the MSAT ID to embed this information within the image, which preserved data integrity by eliminating file naming errors. Images were scanned at 300 dots per inch (dpi) resolution and saved in tagged image file format (TIFF), which is a common minimum recommendation for photo archiving.[10] Test coupons were visually assigned either an adhesive, cohesive, or mixed-mode of failure.

2.4 Dynamic Mechanical Analysis

The bulk adhesive rectangular sample bars made during the bonding process were sanded to uniform thickness using 60-grit followed by 180-grit sandpaper. The samples were cut to a length of approximately 35 mm with a wet saw using sections of adhesive that were visually uniform and free of voiding defects. The thickness and width of a sample were measured using a telescoping micrometer before tightening it in the DMA clamps with a torque screwdriver set to 0.8 Nm (Newton meters). DMA was performed at a constant frequency of 1 Hz and a constant strain of 0.2%. The sample was equilibrated at 0 °C and held isothermally for 10 min, heated to 93 °C at a rate of 2 °C/min, and cooled to RT. Once the first heating cycle was completed, the DMA clamps were retightened, and the sample was cycled a second time. DMA testing yielded the storage modulus (E′), loss modulus (E″). Loss tangent (tan δ) is defined as E″/E′, with the peak taken as the adhesive's glass transition temperature (T_g).

3. Results

The Appendix provides an index of references and links to National Institute of Standards and Technology (NIST) experimental data. Complete records of test data and failure surface scans may be accessed at NIST DSpace repository site (http://hdl.handle.net/11256/939).

3.1 RT (Dry Conditioning)

Figure 2 shows the RT load versus displacement response for the 9309 bonded single lap joints with GPS as the aluminum surface pretreatment. Displacement at failure ($d_{failure}$) and S_{max} are summarized in Table 2, with average values of 34.6 MPa and 3.04 mm, respectively. Visual inspection of the mode of failure was primarily adhesive with a lesser degree of cohesive/adhesive mixed-mode. A representative failure surface shown in Fig. 3.

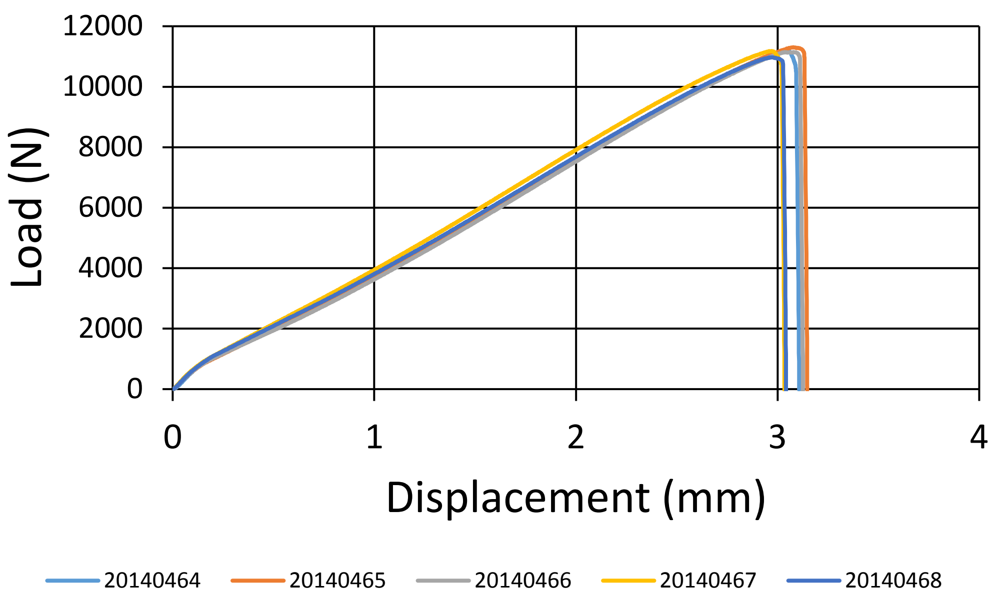

Fig. 2 Load vs. displacement for RT (no conditioning) samples

Table 2 S_{max}, $d_{failure}$, and mode of failure: RT (no conditioning), adhesive (ADH), cohesive (COH), and mixed-mode (MM)

Sample ID	S_{max} (MPa)	$d_{failure}$ (mm)	Mode of failure
20140464	34.7	3.11	ADH/MM
20140465	35.0	3.08	ADH/MM
20140466	34.6	3.05	ADH/MM
20140467	34.7	2.97	ADH/MM
20140468	34.0	2.97	ADH/MM
Avg.	34.6	3.04	. . .
Std dev.	±0.4	±0.06	. . .

Fig. 3 Failure surface for RT (no conditioning) samples. MSAT ID = 20140464, mode of failure = adhesive/mixed-mode.

3.2 RT (Hot/Wet Conditioning)

Figure 4 shows the RT load versus displacement response for the 9309 bonded single lap joints with GPS as the aluminum surface pretreatment following water immersion for 14 days at 63 °C. S_{max} and d_{failure} are summarized in Table 3, with average values of 31.1 MPa and 2.48 mm, respectively. Visual inspection of the mode of failure was adhesive with a representative failure surface shown in Fig. 5.

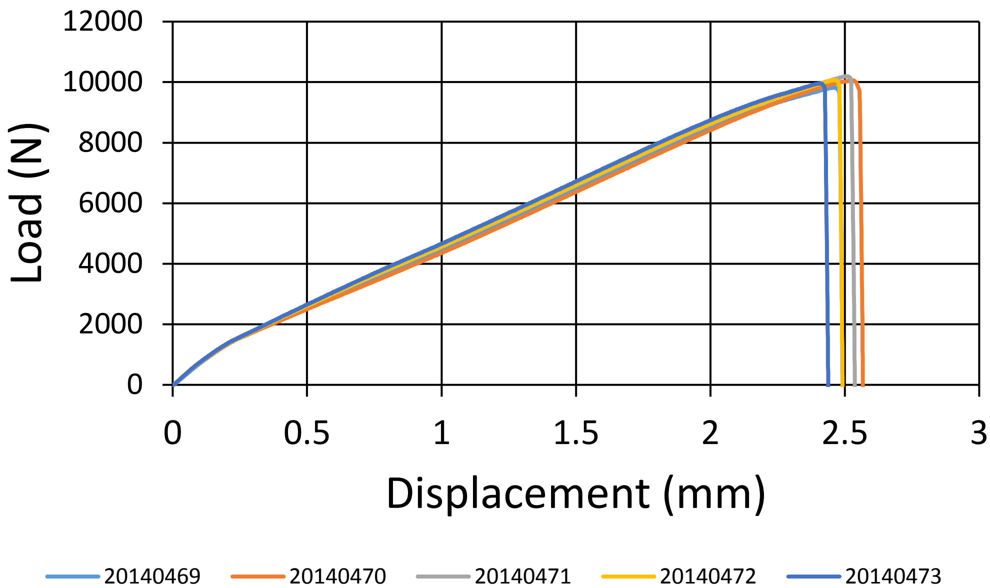

Fig. 4 Load vs. displacement for RT (hot/wet conditioning) samples

Table 3 S_{max}, $d_{failure}$, **and mode of failure: RT (hot/wet conditioning), adhesive (ADH), cohesive (COH), and mixed-mode (MM)**

Sample ID	S_{max} (MPa)	$d_{failure}$ (mm)	Mode of failure
20140469	30.5	2.46	ADH
20140470	31.2	2.53	ADH
20140471	31.6	2.51	ADH
20140472	31.2	2.46	ADH
20140473	30.9	2.44	ADH
Avg.	31.1	2.48	. . .
Std dev.	±0.4	±0.04	. . .

Fig. 5 Failure surface for RT (hot/wet conditioning) samples. MSAT ID = 20140469, mode of failure = adhesive.

3.3 Elevated Temperature

Figure 6 shows the ET load versus displacement response for the 9309 bonded single lap joints with APS as the aluminum surface pretreatment following ET testing at 71 °C. S_{max} and $d_{failure}$ are summarized in Table 4, with average values of 5.6 MPa and 0.78 mm, respectively. Visual inspection of the mode of failure was adhesive with a representative failure surface shown in Fig. 7.

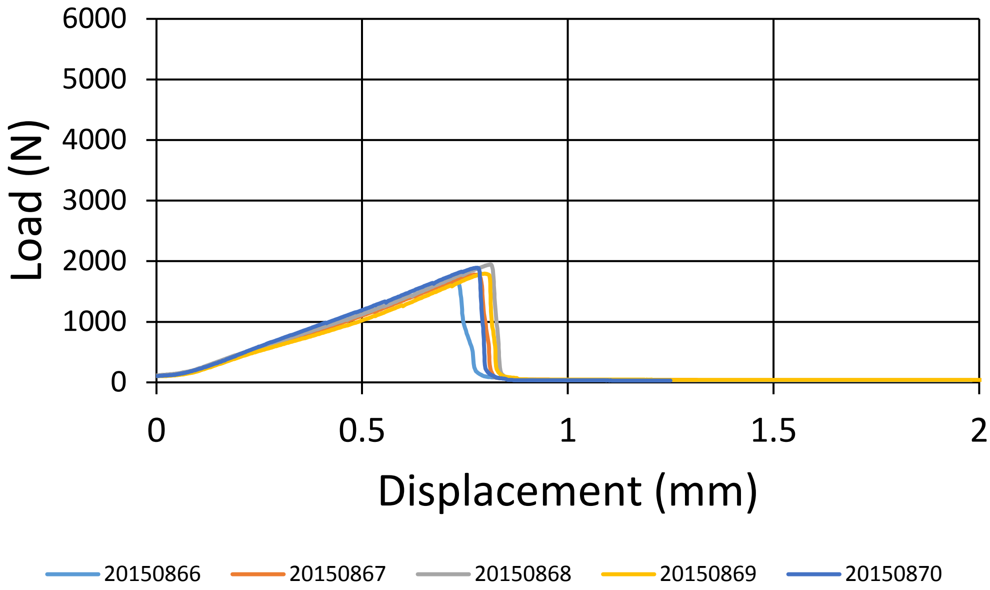

Fig. 6 Load vs. displacement for ET samples (66 °C postcure)

Table 4 S_{max}, $d_{failure}$, and mode of failure: ET (66 °C postcure), adhesive (ADH), cohesive (COH), and mixed-mode (MM)

Sample ID	S_{max} (MPa)	$d_{failure}$ (mm)	Mode of failure
20150866	5.2	0.73	ADH
20150867	5.5	0.77	ADH
20150868	6	0.81	ADH
20150869	5.6	0.80	ADH
20150870	5.9	0.78	ADH
Avg.	5.6	0.78	. . .
Std. dev.	±0.3	±0.03	. . .

Fig. 7 Failure surface for ET sample (66 °C postcure). MSAT ID = 20150866, mode of failure = adhesive.

4. Discussion

Figure 8 shows a plot of S_{max} versus $d_{failure}$ for the 9309 tiered testing against ARL-ADHES-QA-001.00 rev 1.0. The adhesive's strength and displacement performance fits within the Group II defined region under RT testing conditions with no prior environmental exposure. Following hot/wet conditioning by water immersion for 14 days at 63 °C, S_{max} and $d_{failure}$ decrease, but remain within Group II. Under ET test conditions at 71 °C, S_{max} decreases to below the 10.0 MPa threshold of Groups I, II, and III.

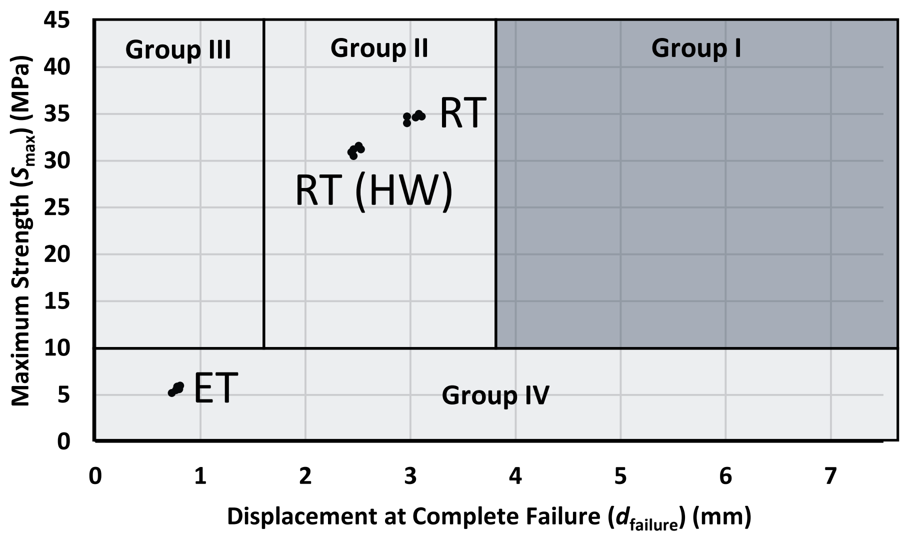

Fig. 8 Hysol EA 9309NA epoxy adhesive property categories as defined by ARL-ADHES-QA-001.00 rev 1.0 (all test conditions). Group I S_{max} ≥ 10.0 MPa and $d_{failure}$ > 3.81 mm, Group II S_{max} ≥ 10.0 MPa and 1.60 mm ≤ $d_{failure}$ ≤ 3.81 mm, Group III S_{max} ≥ 10.0 MPa and $d_{failure}$ < 1.60 mm, Group IV S_{max} < 10.0 MPa. RT = Tier 1 RT, RT (HW) = Tier 2 RT (hot/wet conditioning), ET = Tier 2 ET

The manufacturer's claimed S_{max} for 9309 as 34.5 MPa at RT and 6.9 MPa at 71 °C.[11] The results obtained for this research were 34.6 and 5.6 MPa at room and ET, respectively. Hot/wet testing showed a decrease in S_{max} to 31.1 MPa for a retention of approximately 90% of its dry strength. The decrease in strength under hot/wet conditions is most likely due to hydrolysis of the aluminum/adhesive interface, not the adhesive, and is mitigated by the silane coupling agent pretreatment.[12] At ET the loss of strength is due to testing the adhesive near its T_g. APS was used as the surface pretreatment for the ET test samples, compared with GPS for the RT and hot/wet samples. However, APS has been reported to offer higher interfacial fracture energy on epoxy bonded joints.[13] Therefore, it is unlikely that the APS surface pretreatment resulted in the strength loss at ET.

Figure 9 shows the cycled first and second heating DMA response curves of tan δ versus temperature for 9309. During the first heating DMA cycle the T_g of the adhesive is 79 °C. The T_g increases to 87 °C upon the second heating cycle in the DMA due to additional postcuring of residual unreacted epoxy and curing agent. While the elevated test temperature of 71 °C is below the peaks of the tan δ curves, the distribution of the glass transition region is approached. The additional postcuring of the adhesive through the DMA heating cycles shifts the glass transition region to higher temperatures. Figure 10 shows the decrease in modulus (E′) through the glass transition region, with the adhesive clearly becoming more rubbery during the first DMA heating cycle at 71 °C, which will decrease single-lap-joint S_{max} at this temperature.

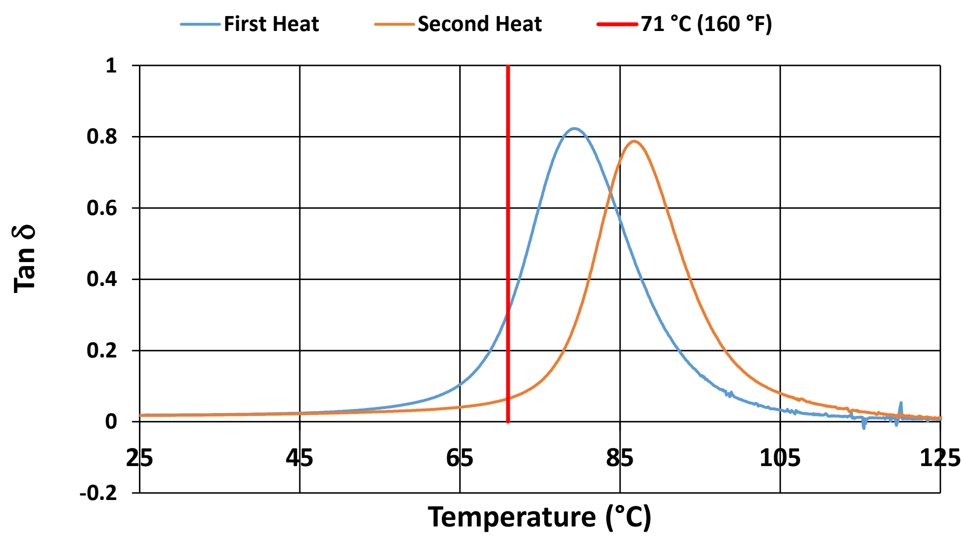

Fig. 9 Plots of tan δ vs. temperature for Hysol EA 9309NA for first and second heat DMA cycles

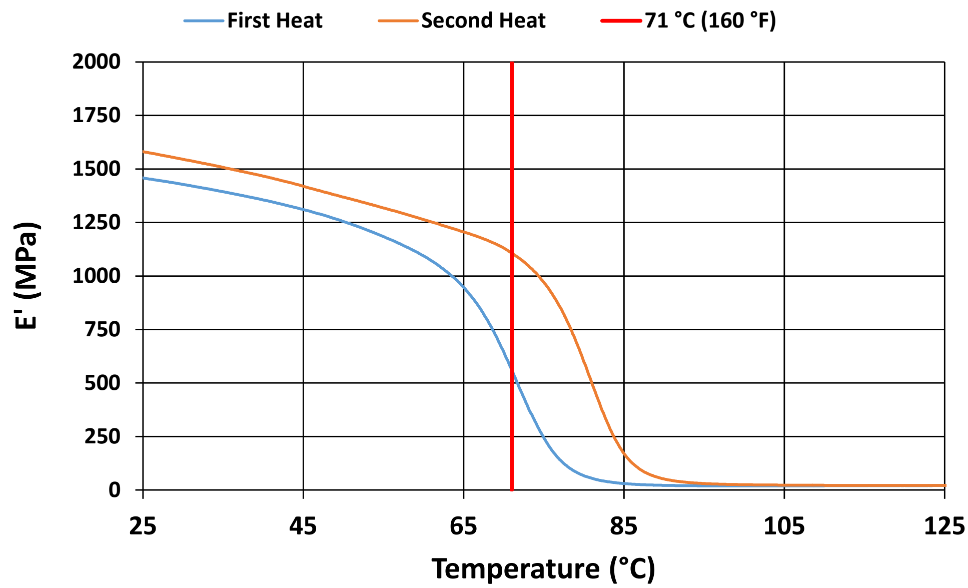

Fig. 10 Plots of E′ vs. temperature for Hysol EA 9309NA for first and second heat DMA cycles

The adhesive postcure of 1 h at 66 °C used for this research is provided as a cure schedule guidance in the manufacturer's technical data sheet, but increased cure temperatures up to 93 °C are also sanctioned.[11] The DMA results clearly show that the adhesive is not fully cured after being held isothermally at 66 °C for 1 h. Incomplete cure under these conditions is likely due to the adhesive vitrifying into a glass and quenching further chemical reactions between the epoxy resin and amine curing agent.[14–17]

To test this hypothesis of vitrification during cure a second set of single lap joints for ET testing was prepared with a postcure cycle of 93 °C for 2 h. Figure 11 shows the load versus displacement response for the 9309 bonded single lap joints with APS as the aluminum surface pretreatment following ET testing at 71 °C. S_{max} and d_{failure} are summarized in Table 5. Visual inspection of the mode of failure was adhesive with a representative failure surface shown in Fig. 12. S_{max} increased from 5.6 to 15.4 MPa when the postcure temperature was increased from 66 to 93 °C, respectively. Failure displacement for the samples postcured at 93 °C increased from 0.78 to 1.23 mm.

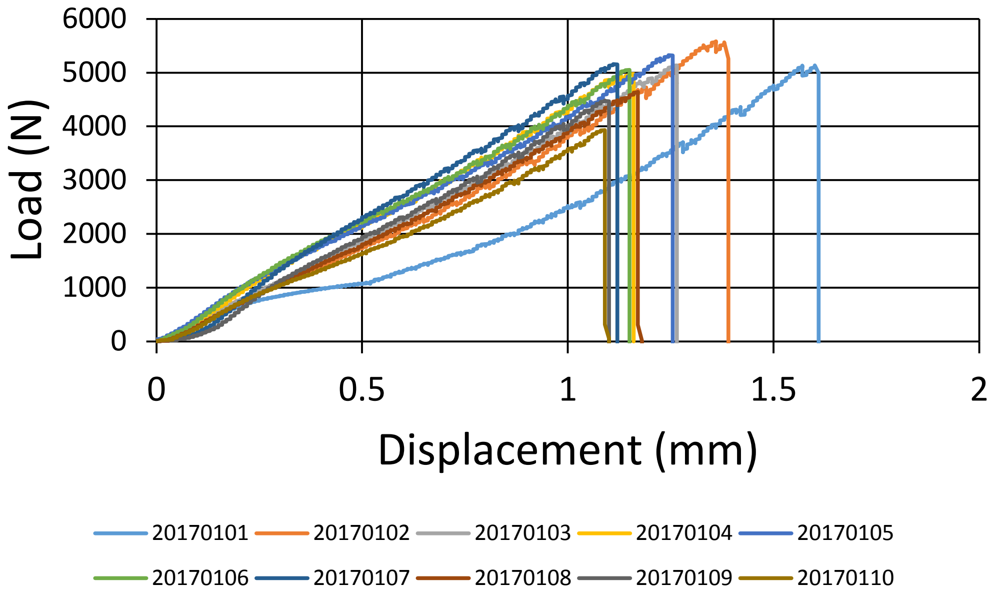

Fig. 11 Load vs. displacement for ET samples (93 °C postcure)

Table 5 S_{max}, $d_{failure}$, and mode of failure: ET (93 °C postcure), adhesive (ADH), cohesive (COH), and mixed-mode (MM)

Sample ID	S_{max} (MPa)	$d_{failure}$ (mm)	Mode of failure
20170101	15.9	1.61	ADH
20170102	17.3	1.39	ADH
20170103	15.9	1.27	ADH
20170104	15.4	1.16	ADH
20170105	16.5	1.26	ADH
20170106	15.6	1.15	ADH
20170107	16.0	1.12	ADH
20170108	14.8	1.18	ADH
20170109	13.9	1.10	ADH
20170110	12.2	1.10	ADH
Avg.	15.4	1.23	. . .
Std. dev.	±0.3	±0.16	. . .

Fig. 12 Failure surface for ET sample (93 °C postcure). MSAT ID = 20170101, mode of failure = adhesive.

The test results for 9309 provide for a reasonable perspective for evaluating the stringency of ARL-ADHES-QA-001.00 rev 1.0, particularly with respect to the second tier hot/wet and ET requirements (Fig. 13). For hot/wet testing S_{max} decreased from 34.6 MPa to 31.1 MPa for a retention of 90% of its dry RT strength. For ET testing S_{max} decreased from 34.6 MPa to 15.4 MPa for a retention of 44% of its dry RT strength. ARL-ADHES-QA-001.00 rev 1.0 calls for strength retention of 75% for both hot/wet conditioning and ET testing.

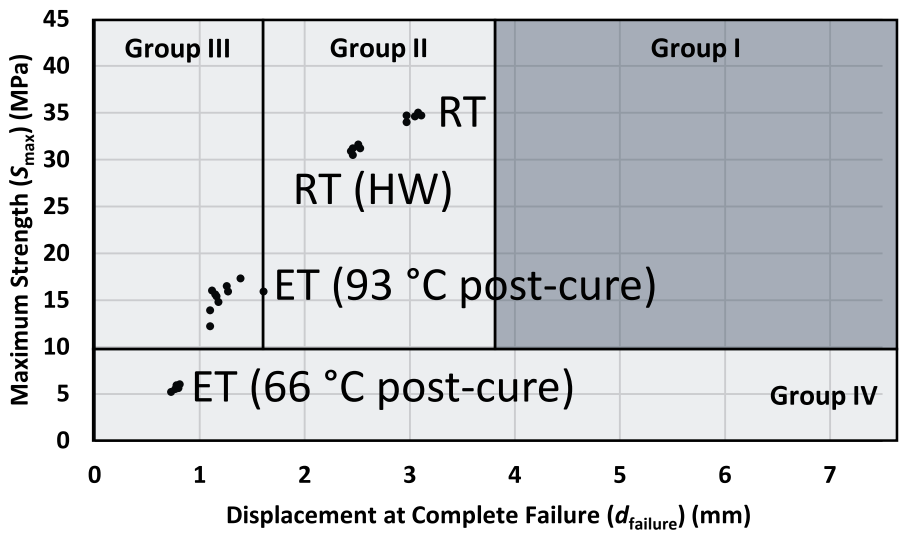

Fig. 13 Hysol EA 9309NA epoxy adhesive property categories as defined by ARL-ADHES-QA-001.00 rev 1.0 (all test conditions and including 93 °C postcure). Group I $S_{max} \geq 10.0$ MPa and $d_{failure} > 3.81$ mm, Group II $S_{max} \geq 10.0$ MPa and $1.60 \text{ mm} \leq d_{failure} \leq 3.81$ mm, Group III $S_{max} \geq 10.0$ MPa and $d_{failure} < 1.60$ mm, Group IV $S_{max} < 10.0$ MPa. RT = Tier 1 RT, RT (HW) = Tier 2 RT (hot/wet conditioning), ET = Tier 2 ET.

For comparison, Table 6 lists the hot/wet conditioning and ET single-lap-joint strength retentions for a variety of structural adhesive performance standards. A majority of the surveyed standards require a minimum hot/wet strength retention of 95%. The 90% hot/wet strength retention for the 9309 is of minimal concern as the single lap joints were pretreated with a silane coupling agent. The standards that cite a minimum of 95% hot/wet strength retention were derived for aerospace applications, where more durable phosphoric acid anodization surface pretreatments are often employed.[18] The hot/wet testing minimum strength retention requirement of 75% for ARL-ADHES-QA-001.00 rev 1.0 is lenient for an epoxy-based adhesive when compared with other established standards. ARL-ADHES-QA-001.00 rev 1.0 does not specify the adhesive chemistry, thus the hot/wet strength retention requirements could be more difficult to obtain for alternative adhesive types.

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Table 6 Hot/wet conditioning (HW) and elevated temperature (ET) single-lap-joint strength retentions for a variety of structural adhesive standards

Specification	RT strength (MPa)	HW strength (MPa)	HW retention (%)	ET strength (MPa)	ET retention (%)
ARL-ADHES-QA-001.00 rev 1.0[a]	≥10.0 (Group I, II, III)	Retain 75% of RT strength	75	Retain 75%	75
A-A-50272[b]	11.0	Not specified	. . .	Not specified	. . .
AMS3686A[c]	16.5	15.9	96.4	11.7 (260 °C)	70.9
AMS3686A[c]	16.5	15.9	96.4	8.3 (315 °C)	50
AMS3692C[d]	13.8	Not specified	. . .	6.9 (260 °C)	50
AMS3695/1[e]	29.0	Change not greater than 5%	95	17.2 (95 °C)	59.3
AMS3695/3[f]	20.1	Change not greater than 5%	95	17.2 (175 °C)	85.6
AMS3695/4[g]	19.3	Change not greater than 5%	95	9.7 (215 °C)	50
ASTM D6412[h]	17.2 (Type I), 8.2 (Type II)	Not specified	. . .	2.8 (I), 1.42 (II) (82 °C)	16 (I), 17 (II)
MMM-A-132B[i]	38 (Class I)	31 (Class I)	81	19 (I) (180 °F)	50 (I)

Notes: [a]ARL-ADHES-QA-001.00 rev 1.0. Jensen R, DeSchepper D, Flanagan D, Kosik-Chaney W, Robinette J, Chaney G, and Pergantis, C. Adhesives: test method, group assignment, and categorization guide for high-loading-rate. ARL-ADHES-QA-001.00, rev 1.0. Aberdeen Proving Ground (MD): Army Research Laboratory (US); 2014 June. Report No.: ARL-SR-0288.

[b]A-A-50272. Adhesives, epoxy, general services administration, commercial item description. Picatinny Arsenal (NJ): Armament Research, Development and Engineering Center (US); 1997 Oct 28. Defines operating temperature range up to 150 °C, but does not define strength requirements.

[c]AMS3686A. Adhesive, polyimide resin, film and paste high temperature resistant, 315 °C (599 °F). Warrendale (PA): SAE International; 2008.

[d]AMS3692C. Adhesive compound, epoxy resin high temperature application. Warrendale (PA): SAE International; 2014.

[e]AMS3695/1. Adhesive film, epoxy-base, high durability for 95 °C (200 °F) service. Warrendale (PA): SAE International; 2014.

[f]AMS3695/3. Adhesive film, epoxy-base, high durability for 175 °C (350 °F) service. Warrendale (PA): SAE International; 2014.

[g]AMS3695/4. Adhesive film, epoxy-base, high durability for 215 °C (420 °F) service. Warrendale (PA): SAE International; 2014.

[h]ASTM D6412/6412M-99. Standard specification for epoxy (flexible) adhesive for bonding metallic and nonmetallic materials. West Conshohocken (PA): ASTM International; 2012, doi: 10.1520/D6412_D6412M-99R12. www.astm.org.

[i]MMM-A-132B. Federal specification: adhesives, heat resistant, airframe structural, metal to metal. Washington (DC): Department of Defense (US); 1994 Apr 1.

However, the elevated strength retention requirement of 75% for ARL-ADHES-QA-001.00 rev 1.0 is stricter than a majority of the aviation derived adhesive standards where 50% is a typical norm. The ET strength retention of 44% for 9309 was obtained by testing just a few degrees Celsius within the onset of the glass transition of the adhesive. The 9309 adhesive would likely have retained 50% of its ET strength at 65 °C, based on the plot of E′ versus temperature shown in Fig. 10. Regardless of the ET performance of 9309, the elevated strength retention requirement of 75% for ARL-ADHES-QA-001.00 rev 1.0 is too high. ARL-ADHES-QA-001.00 rev 1.0 is intended as a screen for rapidly assessing promising adhesives, thus decreasing the elevated strength retention requirement to 50% should enable a broader selection of potential candidates.

5. Conclusions

Hysol EA 9309NA 2-part epoxy paste adhesive was evaluated per ARL-ADHES-QA-001.00 rev 1.0, which consists of single-lap-joint testing at RT and subsequent hot/wet and ET conditioning. RT strength was 34.6 MPa, which decreased to 90% (31.1 MPa) following hot/wet conditioning and 44% (15.4 MPa) when tested at ET. Displacement at failure at RT was 3.04 mm, which falls within the Group II domain of ADHES-QA-001.00 rev 1.0. The screening protocol also recommends adhesive strength retention of 75% of RT dry maximum strength after second tier hot/wet and ET testing to be considered for more in depth analysis. Compared with a survey of commercial and Department of Defense–accepted adhesive performance specifications, the strength retention requirement of 75% following hot/wet conditioning is most likely adequate as a screening threshold. However, 75% strength retention for ET testing is too strict and could be reduced to 50%.

6. References

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Appendix. Supporting Digital File Archive Index

Table A-1 provides the reader with a reference list and URL links to experimental data and supporting metadata descriptors archived in the National Institute of Standards and Technology (NIST) DSpace repository (http://hdl.handle.net/11256/939). Summarized data are attached to this PDF in Microsoft Excel format.

Table A-1 Supporting data collection

File name[a]	Description
Room temperature	Size: 15.76 MB Format: Microsoft Excel, TIFF, PDF Lab notes, load versus displacement data, and failure surface images
Hot-wet	Size: 17.65 MB Format: Microsoft Excel, TIFF, PDF Lab notes, load versus displacement data, and failure surface images
Elevated temperature	Size:78.19 MB Format: Microsoft Excel, TIFF, PDF Lab notes, load versus displacement data, and failure surface images
Dynamic mechanical analysis	Size: 3.394 MB Format: Microsoft Excel and csv text DMA storage and loss modulus
Materials pedigree	Size: 299.4 KB Format: PDF and Microsoft Excel Manufacturer technical and safety data sheets, current and pending environmental legislations
Calibration certs	Size: 9.039 MB Format: PDF Calibration certificates for test equipment used

[a] Abbreviated file name as it appears on NIST site.

List of Symbols, Abbreviations, and Acronyms

A	bonded area
ADH	adhesive mode of failure
APS	3-aminopropyltrimethoxysilane
ARL	US Army Research Laboratory
CAS	chemical abstract service
COH	cohesive mode of failure
CQL	College Qualified Leaders
DMA	dynamic mechanical analysis
dpi	dots per inch
E′	storage modulus
E″	loss modulus
ET	elevated temperature
ID	sample identification number
min	minute
mm	millimeter
MM	mixed-mode mode of failure
MPa	megapascal
MSAT	Materials Selection and Analysis Tool
Nm	Newton meter
NIST	National Institute of Standards and Technology
P_{max}	maximum load
PDF	portable document format
RT	room temperature
S_{max}	maximum strength
T_g	glass transition temperature

tan δ	loss tangent
TIFF	tagged image file format
URL	uniform resource locator

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2 (PDF)	DIRECTOR US ARMY RESEARCH LAB RDRL CIO L IMAL HRA MAIL & RECORDS MGMT
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